(12) UK Patent Application (19) GB (11) 2 121 069 A

(21) Application No 8313474
(22) Date of filing
16 May 1983
(30) Priority data
(31) 1924/82
(32) 17 May 1982
(33) Austria (AT)
(43) Application published
14 Dec 1983
(51) INT CL³ C08L 1/00
C08J 5/02
(52) Domestic classification
C3V AQ
C3W 121
U1S 1596 3020 C3V
(56) Documents cited
None
(58) Field of search
C3V
(71) Applicant
Chemiefaser Lenzing
Aktiengesellschaft
(Austria)
4860 Lenzing
Austria
(72) Inventors
Ingo Marini
Hans Krassig
Jurgen Lenz
Hartmut Ruf
Josef Schmidtbauer
(74) Agent and/or Address for
Service
Page White and Farrer
5 Plough Place
New Fetter Lane
London EC4A 1HY

(54) Cellulose-based fibres for the production of non-wovens

(57) Cellulose fibres having a low water retention capacity and a low water holding capacity as well as a high immersion time, and that are processable into a non-woven exhibiting as short a droplet penetration time as possible, as low a wet retention as possible and as slight a wetback as possible, contain, (a) as mineral fillers, barium sulphate, talcum, muskovite, or a mixture thereof, in an amount of from 15 to 60% of the total fibre mass, and, if desired, (b) additionally, hydrophobic, polymer or oligomer substances, such as polyethylene, polypropylene, polystyrene, polyacrylic acid ester, polyester, polytetrafluorethylene or waxes, in an amount of from 1 to 60% of the total fibre mass. The fibres are produced in a process in which the mineral fillers and, if desired, the hydrophobic, polymer or oligomer substances—preferably as an aqueous suspension of the same—are admixed to a viscose, and the mixture is wet-spun.

GB 2 121 069 A

SPECIFICATION

Improvements in or relating to cellulose-based fibres, in particular for the production of non-wovens, a nonwoven comprising such fibres, and a method of producing the fibres

The invention relates to cellulose-based fibres having a low water retention capacity, a high immersion time, a low water holding capacity (total capacity), a low absorbent capacity, and a content of mineral fillers, in particular for the production of nonwovens, a nonwoven comprising such fibres, as well as a method of producing such fibres.

Fibres of this kind are primarily used in cover stock for children's napkins, adults' napkins, sanitary towels, incontinence towels, bedding linens and similar disposable articles, as well as in padding nonwovens.

It is known, for instance, for Swiss patent No. 487,641 to use carded, binder-containing nonwovens of viscose and/or polyester staple fibres for cover stock. In recent times, binding-fibre-bonded nonwovens of viscose, polyester and/or polypropylene staple fibres have gained more and more importance. According to recent findings, synthetic-fibre-spun nonwovens are intended to be used to a greater extent for the above-mentioned field of application.

In addition to the necessary mechanical data of non-wovens, such as dry and wet tenacities, energy at break, bursting strength, stiffness, etc., special hydrophobic properties have recently been demanded for cover stock. Based on a number of published works presented in the course of lectures by EDANA (European Disposables and Non-wovens Association), the following three measurable variables of cover stock are used to determine these special properties:

—droplet penetration time
—wet retention
—wetback (stain test).

The droplet penetration time is a measure for the primary wetting of the cover stock and is measured in seconds.

The wet retention indicates the amount of liquid in %, based on the weight of the dry cover stock, which is absorbed and retained by the cover stock under the test conditions.

With wetback, the liquid discharge by the cover stock to a superimposed blotting paper is determined and as a measured value the stain area is in dicated in cm^2.

On the basis of these test methods for the characterisation of the hydrophobic properties, as short a droplet penetration time as possible, as low a wet retention as possible and as slight a wetback as possible are required for cover stock. In general, these conditions are better complied with by synthetic fibres than, for instance, by viscose fibres, with respect to wet retention and wetback. In contrast, the primary wetting, which is reflected in a short droplet penetration time, is clearly more favourable with viscose fibres than with synthetic fibres.

The invention has as its object to provide cellulose-based fibres, such as viscose fibres, of the initially described kind, which, in particular, are processable into a nonwoven that has all the above-defined properties.

This object is achieved according to the invention in that the fibres, as mineral filters, contain barium sulphate, talcum, muskovite, or a mixture thereof, in an amount of from 15 to 60%, preferably 40 to 50%, of the total fibre mass, and, if desired, additionally contain hydrophobic, polymer or oligomer substances, such as polyethylene, polypropylene, polystyrene, polyacrylic acid ester, polyester, polytetrafluorethylene, or waxes, in an amount of from 1 to 60%, preferably 25 to 50%, of the total fibre mass.

The particle size of the mineral fillers contained in the fibres ought not exceed 3 μm. In German patent No. 845, 230 it is suggested to add electroosmotically purified kaolin, preferably in amounts of from 5 to 10%, to a cellulose solution prior to its formation into threads or films, yet the addition of kaolin exclusively serves delustering purposes. As waxes, such of petrochemical, montanistic or synthetic proveniences may be used for the fibres according to the invention.

The portion of mineral fillers and/or polymer or oligomer substances in the fibres according to the invention in the first place dependes on the special fibre properties desired in the sense of a hydrophobic finish.

Advantageously, the fibres contain a mixture of mineral fillers and hydrophobic, polymer or oligomer substances in a mass ratio of between 10:90 and 90:10, preferably between 50:50 and 90:10, with the mixture being particularly contained in an amount of between 1 to 60%, preferably 30 to 50%, of the total fibre mass.

According to a further advantageous embodiment, the fibres contain mineral fillers that are coated with the hydrophobic, polymer or oligomer substances at a mass ratio of between 75:25 and 99:1, preferably between 85:15 and 95:5, wherein these coated mineral fillers in particular are contained in an amount of from 1 to 60%, preferably 25 to 50%, of the total fibre mass.

While the reduction of hydrophility, with the addition of mineral fillers, primarily is based on the fact that the hydrophilic groups of the cellulose forming the fibre structure are reduced in an

economical manner as compared to pure cellulose fibres, the addition of hydrophobic polymer or oligomer substances is a hydrophobic finish induced on purpose. If a mixture of mineral fillers and hydrophobic polymer or oligomer products is added to the viscose, different hydrophobic finish stages are achieved in the resulting fibre according to the invention, depending on the mixing ratio of these two components.

The production of the fibres, according to the invention is effected in that the mineral fillers and, if desired, the hydrphobic, polymer or oligomer substances or the coated mineral fillers—preferably as an aqueous suspension of the same—are admixed to a viscose, and the mixture is wetspun.

Preferably, the mineral fillers or the hydrophobic substances are used as an aqueous suspension with a particle or drop size of below 3 μm, in the presence of an alkali-stable emulgator system. The alkali-stable emulgator system on the one hand prevents the agglomeration of particles after having been stirred into the viscose and on the other hand collapses in the usually sulphuric spin bath, so that the mineral fillers and the finely distributed hydrophobic, polymer or oligomer substances are incorporated into the cellulose fibres possibly without loss.

A nonwoven according to the invention is characterised in that, at a fibre titre of 1.7 dtex, it has a droplet penetration time of no more than 45 s, a wet retention of no more than 90% and a wetback of no more than 55/75 cm^2, or that, at a fibre titre of 3.3 dtex, it has a droplet penetration time of no more than 10 s, a wet retention of no more than 45% and a wetback of nor more than 40/65 cm^2.

The invention will now be explained in more detail by way of the following examples.

The measurable variables necessary to characterise the fibre properties are defined as follows:

$$\text{Thickness swelling [\%]} = \frac{D_{\text{swollen fibre}} - D_{\text{cond. fibre}}}{D_{\text{cond. fibre}}} \times 100$$

D: fibre diameter

The thickness swelling is determined microscopically at an individual fibre and represents the diameter increase of a fibre swollen in water, based on the conditioned fibre.

$$\text{Steam absorption [\%]} = \frac{\text{fibre mass at 25°C and 90\% rel. humidity} - \text{fibre mass in the absolutely dry state}}{\text{fibre mass in the absolutely dry state}} \times 100$$

The fibres are stored at the indicated temperature and relative humidity until the condition of equilibrium has been attained. The water absorbed is gravimetrically determined.

The immersion time and the water holding capacity (WHV) are methods of determination described in the Deutschen Arzneibuxh (DAB), 7th Ed., 1968. The immersion time is a measure for the wettability of the fibres and is indicated in seconds, a good wettability being indicated by a short immersion time. The water holding capacity indicates the water held in the capillary tubes between the fibres in g/g of fibre.

The water retention capacity (WRV) according to DIN 53814 is a measure for the water retained in the individual fibres after intensive immrsion in water and subsequent defined centrifugation and is indicated in %.

$$\text{WRV [\%]} = \frac{\text{fibre mass centrifuged} - \text{fibre mass dry}}{\text{fibre mass dry}} \times 100$$

The absorbent capcity is determined according to the Demand-Wettability-Test, published by B.M. Lichstein, 2nd Annual Symposium on Nonwoven Product Development, March 1974, Washington, D.C., 129 to 142.

Example 1:

Into 1 kg of viscose containing 8 to 9% cellulose, 5 to 5.5% NaOH and 2 to 2.5% sulphur, 115 g of an aqueous suspension having a content of 30% barium sulphate (Blanc Fixe Micro from Sachtleben) were stirred.

This mixed viscose was spun in a spin bath having a content of 100 to 110 g of H_2SO_4, 340 to 360 g of Na_2SO_4 and 5 to 10 g of $ZnSO_4$ per l. The spin bath temperature was 40 to 50°C. The coagulated fibre cable was drawn by 50% in air, cut into staples, washed, bleached, avived and dried.

Fibre properties:

Titre (dtex)/staple length (mm)	1.7/40
Fibre tenacity conditioned (cN/tex)	12.2
Fibre elongation conditioned (%)	15.0
Degree of whiteness (BE)	65.0
Barium sulphate content (%)	28.0
WRV according to DIN 53814 (%)	63.5
Immersion time according to DAB 7 (s)	3.5
WHV according to DAB 7 (g/g)	16.5
Absorbent capacity according to Demand-Wettability-Test (ml/g)	8.1

On the addition of 30% of a thermoplastic Heterofil binding fibre (CHISSO ES), the fibres were carded four times and thermally strengthened at about 200°C to obtain a uniform distribution.

Nonwoven properties:

Droplet penetration time (s)	0
Wet retention (%)	55.0
Wetback (cm^2)	53/75

Without avivation, the fibres exhibit a very high fibre-fibre cohesion, which is expressed by a high mean maximum cohesion of the sliver and a high value for the range of cohesion and slipping:

Mean maximum cohesion of the silver (cN/ktex)	45.8 to 62.1
Range of cohesion and slipping (cN/ktex)	16.8

On account of this high fibre-fibres cohesion it was possible to produce ear sticks from the fibres of the invention. Usually, only the highly cohesive cotton has been used for the production of ear sticks.

The fibre according to the invention also exhibits the property of being embossable, which is demanded for the production of cotton round pads for cosmetics. Round pads are punched out of a nonwoven by means of a special tool, the round pads having to have a characteristic embossed edge in order to largely prevent fibres from fuzzling off during utilisation of the round pads. Usually, only natural cotton exhibits this characteristic property.

Example 2:

It is proceeded in a manner analogous to Example 1, yet in addition to the barium sulphate suspension, 88g of an aqueous emulsion having a content of 39% polyethylene (Polyäthylen-Dispersion PE 30 from Hoechst) are stirred into the viscose.

Fibre properties:

Titre (dtex)/staple length (mm)	1.7/40	3.3/60
Fibre tenacity cond. (cN/tex)	10.3	9.9
Fibre elongation cond. (%)	10.5	11.5
Degree of whiteness (BE)	61.0	63.0
Barium sulphate content (%)	27.8	27.5
Polyethylene content (%)	18.2	18.9
WRV according to DIN 53814 (%)	57.5	52.3
Immersion time according to DAB 7 (s)	6.0	5.7
WHV according to DAB 7 (g/g)	15.0	15.3
Absorbent capacity according to Demand-Wettability-Test (ml/g)	7.5	7.8

Nonwoven properties:

Droplet penetration time (s)	2	1
Wet retention (%)	51	49
Wetback (cm^2)	48/65	43/68

Example 3:

According to Example 1, yet with the addition of 128 g of an aqueous dispersion having a content of 30% of a wax-coated barium sulphate (90% Blanc Fixe Micro from Sachtleben and 10% MOBILCER 46 from Mobil) instead of the barium sulphate suspension, fibres and a

nonwoven were obtained.

Fibre properties:		
Titre (dtex)/staple length (mm)	1.7/40	3.3/60
Fibre tenacity cond. (cN/tex)	10.5	10.1
Fibre elongation cond. (%)	10.2	11.0
Degree of whiteness (BE)	64.0	65.0
Barium sulphate content (%)	27.5	28.0
Portion extractable with petroleum ether (%)	3.5	3.7
WRV according to DIN 53814 (%)	68.9	44.3
Immersion time according to DAB 7 (s)	∞	∞
WHV according to DAB 7 (g/g)	0	0
Absorbent capacity according to Demand-Wettability-Test (ml/g)	0	0
Nonwoven properties:		
Droplet penetration time (s)	43.0	7.0
Wet retention (%)	31.0	24.0
Wetnback (cm^2)	7/34	26/57

Example 4:

According to Example 1, yet with the addition of 115 g of an aqueous suspension having a content of 30% of a talcum (Naint talcum V 118/2) instead of the barium sulphate suspension, fibres and a nonwoven having the following properties were produced.

Fibre properties:	
Titre (dtex)/staple length (mm)	3.3/60
Fibre tenacity cond. (cN/tex)	10.5
Fibre elongation cond. (%)	12.0
Degree of whiteness (BE)	55.0
Talcum content (%)	27.3
WRV according to DIN 53814 (%)	74.2
Immersion time according to DAB 7 (s)	1.9
WHV according to DAB 7 (g/g)	16.2
Absorbent capacity according to Demand-Wettability-Test (ml/g)	8.3
Nonwoven properties:	
Droplet penetration time (s)	0
Wet retention (%)	59
Wetback (cm^2)	42/65

Example 5:

According to Example 1, yet with the addition of 57 g of an aqueous dispersion having a content of 25% methylmethacrylate and 25% butylacrylate (Acrylatdispersion AA 37 from Hoechst) as well as of 70 g of the barium sulphate suspension according to Example 1, fibres and a nonwoven were produced.

Fibre properties:	
Titre (dtex)/staple length (mm)	3.3/60
Fibre tenacity cond. (cN/tex)	11.0
Fibre elongation cond. (%)	15.0
Degree of whiteness (BE)	56.0
$BaSO_4$ content (%)	17.0
polyacrylate content (%)	13.8
WRV according to DIN 53814 (%)	69.0
Immersion time according to DAB 7 (s)	2.9
WHV according to DAB 8 (g/g)	14.5
Absorbent capacity according to Demand-Wettability-Test (ml/g)	7.4
Nonwoven properties:	
Droplet penetration time (s)	2
Wet retention (%)	53
Wetback (cm^2)	39/61

Comparative Example:

It was proceeded as in Example 1, yet without the addition of barium sulphate.

Fibre properties:		
Titre (dtex)/cut length (mm)	1.7/40	3.3/60
Fibre tenacity cond. (cN/tex)	19.0	18.5
Fibre elongation cond. (%)	18.5	20.1
Degree of whiteness (BE)	63.0	62.0
WRV according to DIN 53814 (%)	85.0	85.0
Immersion time according to DAB 7 (s)	2.5	2.2
WHV according to DAB 7 (g/g)	19.0	19.0
Thickness swelling (%)	37.0	37.0
Steam absorption at 20°C and 90% rel. humidity (%)	21.0	21.0
Absorbent capacity according to Demand-Wettability-test (ml/g)	10.2	10.2

Nonwoven properties:

	Fibre titre (dtex)/staple length (mm)	
	1.7/40	3.3/60
Droplet penetration time (s)	0	0
Wet retention (%)	151.0	65.0
Wetback (cm^2)	73/92	47/71

It can be seen that the conventional viscose fibres obtained according to the Comparative Example, as compared to the fibres of the invention, have a considerably higher WRV, a largely reduced immersion time, a much higher WHV and a higher absorbent capacity than the fibres according to the invention.

Nonwovens produced of the fibres according to the invention, as compared to nonwovens produced of conventional viscose fibres, exhibit a sufficiently short, or equal droplet penetration time, while, at the same time, having considerably reduced values for wet retention and wetback.

CLAIMS

1. Cellulose-based fibres having a low water retention capacity, a high immersion time, a low water holding capacity, a low absorbent capacity, and a content of mineral fillers, in particular for the production of nonwovens, characterised in that they contain, as mineral fillers, barium sulphate, talcum, muskovite, or a mixture thereof, in an amount of from 15 to 60%, preferably 40 to 50%, of the total fibre mass, and, if desired, additionally, hydrophobic, polymer or oligomer substances, such as polyethylene, polypropylene, polystyrene, polyacrylic acid ester, polyester, polytetrafluorethylene or waxes, in an amount of from 1 to 60%, preferably 25 to 50%, of the total fibre mass.

2. Fibres according to claim 1, characterised in that they contain a mixture of mineral fillers and hydrophobic, polymer or oligomer substances at a mass ratio of between 10:90 and 90:10, preferably between 50:50 and 90:10.

3. Fibres according to claim 2, characterised in that the mixture is contained in an amount of from 1 to 60%, preferably 30 to 50%, of the total fibre mass.

4. Fibres according to claim 1, characterised in that they contain mineral fillers coated with the hydrophobic, polymer or oligomer substances at a mass ratio of between 75:25 and 99:1, preferably of between 85:15 and 95:5.

5. Fibres according to claim 4, characterised in that the coated mineral fillers are contained in an amount of from 1 to 60%, preferably 25 to 50%, of the total fibre mass.

6. A nonwoven comprising the fibres according to claims 1 to 5, characterised in that, at a fibre titre of 1.7 dtex, it exhibits a droplet penetration time of no more than 45 s, a wet retention of no more than 90% and a wetback of no more than 55/75 cm^2.

7. A nonwoven comprising the fibres according to claims 1 to 5, characterised in that, at a fibre titre of 3.3 dtex, it exhibits a droplet penetration time of no more than 10 s, a wet retention of no more than 45% and a wetback of no more than 40/65 cm^2.

8. A method of producing the fibres according to claims 1 to 5, characterised in that the mineral fillers and, if desired, the hydrophobic, polymer or oligomer substances or the coated mineral fillers—preferably as an aqueous suspension of the same—are admixed to a viscose, and the mixture is wetspun.

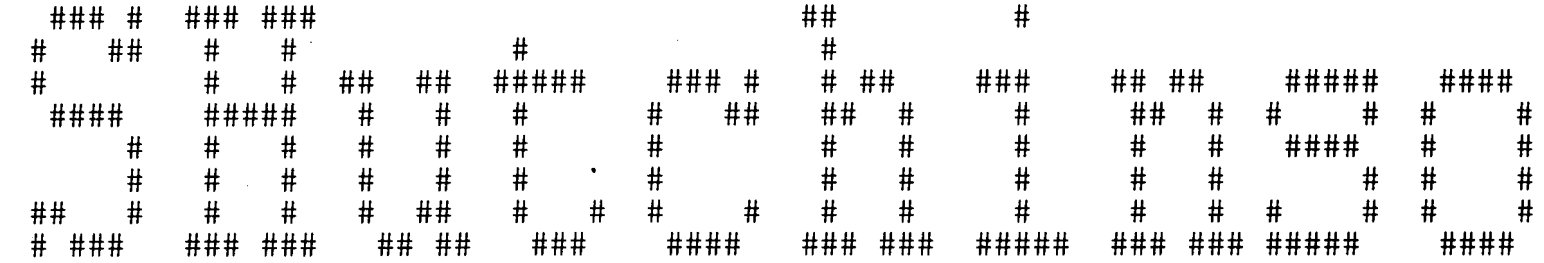
9. A method according to claim 8, characterised in that the mineral fillers and/or hydrophobic substances are used as an aqueous suspension with a particle or droplet size, respectively, of below 3 μm, in the presence of an alkali-stable emulgator system.

10. Fibres substantially as hereinbefore described with reference to the accompanying examples.

11. A nonwoven substantially as hereinbefore described with reference to the accompanying examples.

12. A method substantially as hereinbefore described with reference to the accompanying examples.

Printed for Her Majesty's Stationery Office by Burgess & Son (Abingdon) Ltd.—1983.
Published at The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.



Job : 158
Date: 8/7/2007
Time: 1:06:19 PM